



6560-50-P

## **ENVIRONMENTAL PROTECTION AGENCY**

**[EPA-HQ-RCRA-2012-0072; FRL-9929-37-OSWER]**

### **Waste Management System; Testing and Monitoring Activities; Notice of Availability of Final Update V of SW-846**

**AGENCY:** Environmental Protection Agency (EPA).

**ACTION:** Notice.

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#### **SUMMARY:**

The Environmental Protection Agency (EPA or Agency) is providing notice of the availability of "Final Update V" to the Third Edition of the manual, "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," EPA publication SW-846. Final Update V contains analytical methods, of which 8 are new and 15 are revised. The methods in Update V may be used in monitoring or complying with the Resource Conservation and Recovery Act (RCRA) hazardous waste regulations. This action includes revisions to the methods in response to comments

received on a Notice published in the Federal Register on October 23, 2013 and finalizes the methods and guidance. In addition, the Agency is also finalizing revisions to Chapters One through Five of SW-846 and an Office of Resource Conservation and Recovery (ORCR) policy statement in the SW-846 methods compendium. The Agency is issuing this Update as guidance since the changes in this document to the SW-846 analytical methods are not required by RCRA's hazardous waste regulations. Any required analytical methods have not been changed.

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## **SUPPLEMENTARY INFORMATION:**

### **I. General Information**

#### *A. Does This Action Apply to Me?*

This notice is directed to the public in general. It may, however, be of particular interest to those conducting waste sampling and analysis for RCRA-related activities. This universe might include any entity that generates, treats, stores, or disposes of hazardous or nonhazardous solid waste and might also include any laboratory that conducts waste sampling and analyses for such entities.

#### *B. How Can I Get Copies of Final Update V and Other Related Information?*

1. The Agency has established a docket for this action under Docket ID No. EPA-RCRA-2012-0072; FRL-9901-86-OSWER and FRL- 9929-37-OSWER. Publicly available docket materials are available either electronically through [www.regulations.gov](http://www.regulations.gov) or in hard copy at the OSWER RCRA Docket in the EPA Docket Center (EPA/DC), EPA West, Room 3334, and 1301 Constitution Ave., NW, Washington, DC. The EPA Docket Center Public Reading Room is open from 8:30 a.m. to 4:30 p.m., Monday through Friday, excluding legal holidays. The telephone number for the Public Reading Room is (202) 566-1744, and the telephone number for the OSWER RCRA Docket is (202) 566-0270.

*C. How Can I Get Copies of the Third Edition of SW-846 its Updates?*

The Third Edition of SW-846, as amended by Final Updates I, II, IIA, IIB, III, IIIA, IIIB, IVA, IVB, and V, is available in pdf format on the Internet at <http://www.epa.gov/SW-846>.

*C. How Is the Rest of this Notice Organized?*

Sections:

- II. What is the Subject and Purpose of this Notice?
- III. Why is the Agency Releasing Update V to SW-846?
- IV. What Does Final Update V Contain?
- V. What Revisions Are Discussed in This Notice?
- VI. Summary

**II. What is the Subject and Purpose of this Notice?**

The Agency is announcing publication of Final Update V to “Test Methods for Evaluating Solid Waste, Physical/Chemical Methods,” EPA publication SW-846, which is now part of the SW-846 methods compendium. Specifically, Update V of SW-846 contains revisions to the first five chapters of SW-846 and 23 new and modified analytical methods that the Agency has evaluated, and/or revised and determined to be appropriate and may be used for monitoring or complying with the RCRA hazardous waste regulations. Eight of the 23 methods are new methods that have been fully validated, i.e., they have completed technical and Agency workgroup review and approval. In addition these eight new methods are being announced in the Federal Register through this notice. Since the methods have completed the approval process, they will be removed from the “Validated Methods” link at:

[http://www.epa.gov/epawaste/hazard/testmethods/sw846/new\\_meth.htm](http://www.epa.gov/epawaste/hazard/testmethods/sw846/new_meth.htm) and incorporated in the SW-846 methods compendium at:

<http://www.epa.gov/epawaste/hazard/testmethods/sw846/online/index.htm>.

The 15 revised methods have replaced the previous versions in the final update package and will also be placed into the SW-846 methods compendium. Because the RCRA hazardous waste regulations do not require the analytical methods contained in Update V, the Agency is issuing this update as guidance. This guidance does not add or change the RCRA regulations, and does not have any impact on existing rulemakings associated with the RCRA program. To date, the Agency has finalized Updates I, II, IIA, IIB, III, IIIA, IIIB, IVA, and IVB to the SW-846 manual, which can be found on the Agency’s ORCR Web page at: [http:// www.epa.gov/SW-846](http://www.epa.gov/SW-846).

### **III. Why is the Agency Releasing Final Update V to SW-846?**

SW-846 is revised over time as new information and data become available. The Agency continually reviews advances in analytical instrumentation and techniques and periodically incorporates such advances into SW-846 as method updates by adding new methods to the manual, and replacing existing methods with revised versions of the same method. On October 23, 2013, the Agency published a FR Notice (78 FR 63185), announcing the availability of Update V to SW-846. When the comment period closed on January 23, 2014, the Agency received a total of 111 technical and general comments on the Update. The Agency revised the methods and chapters based on comments received, when it was appropriate to do so.

Revisions made were either editorial for clarity or technical for accuracy. A summary of significant changes are noted in Appendix A of each revised method.<sup>1</sup> In addition, significant revisions to the chapters are discussed in Section V of this Notice. These methods can be used for any RCRA applications, other than those specifically required by regulation. In cases that the regulation does not specify the method, the analyst should select an appropriate method in which the performance can be demonstrated and meet project-specific Data Quality Objectives (DQOs).

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<sup>1</sup> Specifically, this summary of significant changes (Appendix A) is included in each newly-revised method referenced in this notice, to assist users in identifying changes from the prior version of the method. EPA also intends to include such summaries in future method revisions.

On a related matter, the Agency is also finalizing an ORCR Policy Statement that responds to concerns the Environmental Laboratory Advisory Board (ELAB) has expressed regarding the official version and status of various methods. ELAB is a committee established under the Federal Advisory Committee Act (FACA) that advises the Agency on measurement, monitoring, and laboratory science issues. The ELAB contacted the Agency's Forum on Environmental Measurements (FEM)<sup>2</sup> with several issues regarding the use of SW-846, specifically seeking clarification about which versions of a revised method are recommended, and seeking clarification in defining terminology used to identify the category of methods.

The Agency did not receive any comments regarding the content of the ORCR Policy Statement and has finalized it without change. As a reminder, the Agency strongly recommends the use of the latest version of an SW-846 method. The Agency, however, is not imposing restrictions on the use of earlier versions of non-required SW-846 methods or precluding the use of previous guidance, if such use is appropriate. For example, earlier versions of an SW-846 method may be more appropriate for regulatory purposes (e.g., for compliance with an existing permit or consent decree), or when new method versions may be more costly to run or perform, than necessary for meeting project-specific objectives.

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<sup>2</sup> The FEM is a standing committee of senior EPA managers established in 2003 to promote consistency and consensus within the EPA on measurement issues, and provide an internal and external contact point for addressing measurement methodology, monitoring, and laboratory science issues with multi-program impacts.

#### **IV. What Does Final Update V Contain?**

Final Update V contains revisions to Chapters One through Five of EPA's publication SW-846.

As noted above, no changes are made to Method Defined Parameters (MDPs), which are required by the RCRA regulation and must be followed prescriptively. Also, no changes were made to general sections of SW-846 to the extent they apply to MDPs. The analytical methods in Update V are considered guidance, provide a basic standard operating procedure, and may be modified where appropriate.

In addition, included in the original Update V Notice, was "The ORCR Policy Statement," which was developed as a result of stakeholders' discussions regarding a need for clarification of the status and definitions (e.g., validated, final, superseded) of methods in SW-846. For example, the policy statement is clear that "the most recent version" of an approved method in SW-846, should be used, unless an existing permit, consent decree, etc.) This policy statement appeared in the original Update V Federal Register Notice. See: October 23, 2013 (78 FR 63188 – 63190), and has been inserted in SW-846 in the table of contents after the Preface. For more information on the policy statement see:

<http://www.epa.gov/wastes/hazard/testmethods/sw846/online/index.htm>. The Agency further notes that its Quality Assurance/Quality Control (QA/QC) guidance (e.g., lower limit of quantitation (LLOQ), relative standard error (RSE), initial demonstration of proficiency (IDP), etc.), while it appears in Chapter One, is also discussed in appropriate sections of the individual methods. Updated V documents are dated July 2014, even though this Update is announced

publicly in this 2015 Federal Register Notice. The July 2014 documents are identified as “Update V” in the document footer.

Table 1 provides a listing of the five revised chapters and 23 methods in this Update V.

TABLE 1 FINAL UPDATE V (METHODS, CHAPTERS AND GUIDANCE)

Analytical Method No.	Method or chapter title
	Table of Contents
	Chapter One—Quality Control
	Chapter Two—Choosing the Correct Procedure
	Chapter Three—Inorganic Analytes
	Chapter Four—Organic Analytes
	Chapter Five—Miscellaneous Test Methods
1030	Ignitability of Solids
3200*	Mercury Species Fractionation and Quantification by Microwave-Assisted Extraction, Selective Solvent Extraction and/or Solid Phase Extraction
3511*	Organic Compounds in Water by Microextraction
3572*	Extraction of Wipe Samples for Chemical Agents
3620C	Florisil Cleanup
4025*	Screening for Polychlorinated Dibenzodioxins and Polychlorinated Dibenzofurans (PCDD/Fs) by Immunoassay
4430*	Screening for Polychlorinated Dibenzo-p-Dioxins and Furans (PCDD/Fs) by Aryl Hydrocarbon Receptor PCR Assay
4435*	Method for Toxic Equivalent (TEQS) Determination for Dioxin-Like Chemical Activity With the CALUX® Bioassay
5021A	Volatile Organic Compounds in Various Sample Matrices Using Equilibrium Headspace Analysis
6010D	Inductively Coupled Plasma-Atomic Emission Spectrometry
6020B	Inductively Coupled Plasma-Mass Spectrometry
6800	Elemental and Speciated Isotope Dilution Mass Spectrometry



8000D	Determinative Chromatographic Separations
8021B	Aromatic and Halogenated Volatiles by Gas Chromatography Using Photoionization and/or Electrolytic Conductivity Detectors
8111	Haloethers by Gas Chromatography
8270D	Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry
8276*	Toxaphene and Toxaphene Congeners by Gas Chromatography/Negative Ion Chemical Ionization Mass Spectrometry (GC–NICI/ MS)
8410	Gas Chromatography/Fourier Transform Infrared Spectrometry for Semivolatile Organics: Capillary Column
8430	Analysis of Bis(2-Chloroethyl)Ester and Hydrolysis Products by Direct Aqueous Injection
9013A	Cyanide Extraction Procedure for Solids and Oils
9014	Titrimetric and Manual Spectrophotometric Determinative Methods for Cyanide
9015*	Metal Cyanide Complexes by Anion Exchange Chromatography and UV Detection
9320	Radium 228

\*New Method

## V. What Revisions are Discussed in This Notice?

### *A. SW-846 Chapters One through Five and QA/QC Guidance*

SW-846 contains the following 13 chapters, which provide additional guidance when conducting sample collection, preparation, treatment and disposal. The first five chapters were revised and/or updated in accordance with Update V method revisions. All the chapter titles for SW-846 are listed in Table 2.

TABLE 2 SW-846 CHAPTERS

CHAPTER ONE	QUALITY CONTROL
CHAPTER TWO	CHOOSING THE CORRECT PROCEDURE
CHAPTER THREE	INORGANIC ANALYTES

CHAPTER FOUR	ORGANIC ANALYTES
CHAPTER FIVE	MISSCELLANEOUS TEST METHODS
CHAPTER SIX	PROPERTIES
CHAPTER SEVEN	CHARACTERISTIC INTRODUCTION AND REGUALTORY DEFINITIONS
CHAPTER EIGHT	METHODS FOR DETERMINING CHARACTERISTICS
CHAPTER NINE	SAMPLING PLAN
CHAPTER TEN	SAMPLING METHODS
CHAPTER ELEVEN	GROUND WATER MONITORING
CHAPTER TWELVE	LAND TREATMENT MONITORING
CHAPTER THIRTEEN	INCINERATION

The date that the technical workgroup officially updated the methods is also displayed in the footer of Update V methods and chapters. Specifically, discussion of the comments and the Agency's responses follow:

### **Chapter One (Quality Control)**

The Agency received 20 comments on Chapter One. Most comments were favorable. For those that were not, the comments mainly focused on the interpretation of terminology used (e.g., Field Blank, Sensitivity, Limit of Quantitation (LOQ), Reproducibility, etc.). Changes to this terminology have been added to the glossary section. The Agency has revised Chapter One for clarity of terminology. The final guidance is more user friendly and more consistent with the Agency's official guidance on QA/QC implementation and procedures (e.g., Quality Assurance Project Plans (QAPPs), DQOs, and the Flexible Approach to Environmental Measurement),

located at: [http://www.epa.gov/quality/qa\\_docs.html#noneparqt](http://www.epa.gov/quality/qa_docs.html#noneparqt). Revisions were also made to improve and clarify the language on LLOQ and blank contamination. In addition, EPA added and revised several QA/QC concepts in Chapter One. The concepts are now included in Chapter One (Quality Control) and individual methods where appropriate. These changes are described below:

*Lower Limit of Quantitation (LLOQ)* - The Agency received 35 comments on the LLOQ concept. Most comments were favorable. As discussed in the October 2013 Federal Register notice, the Agency recommends establishing the LLOQ as the lowest point of quantitation, which in most cases is the concentration of the lowest calibration standard in the calibration curve that has been adjusted for the preparation mass and/or volume. The LLOQ value is a function of both the analytical method and the sample being evaluated.

The Method Detection Limit (MDL) procedure in 40 CFR Part 136, Appendix B, for the determination of MDLs developed for the Clean Water Act (CWA) program uses a clean matrix (i.e., reagent water for preparing “spiked” samples, or samples with known constituent concentrations). Analytical laboratories often have difficulty demonstrating they can meet the MDL established using Part 136 when evaluating complex matrices, such as wastes (e.g., soils, sludges, wipes, and spent materials). This MDL approach generally yields unrealistic and/or unachievable method detection limits for these complex matrices. Since the current Part 136 procedure is generally not suitable for RCRA wastes or materials encountered under the RCRA program, the Agency has chosen to finalize the LLOQ for SW-846. The procedure outlined in Part 136 is currently under review and is being revised for consideration in a future rulemaking

effort. The LLOQ considers the effect of sample matrix (e.g., components of a sample other than the analyte) by taking the LLOQ sample through the entire analytical process, including sample preparation, clean up (to remove sample interferences), and determinative procedures. Lastly, results above the LLOQ are quantifiable within acceptable precision and bias. Thus, the LLOQ approach better suits the needs of the RCRA program, because it provides reliable and defensible results, especially at the lower level of quantitation, and can be reported with a known level of confidence for the complex matrices being evaluated. The Agency uses MDLs in some of the MDPs and understands that other Agency programs may continue to use MDLs to meet their program use and needs (e.g., the National Pollutant Discharge Elimination System (NPDES) permit program).

Since the current MDL procedure is not suitable for complex matrices found in RCRA waste, references to the MDL have been replaced with the LLOQ for non-regulatory methods (guidance). As the regulations are revised, the RCRA program will remove the MDL reference from the MDPs and replace it with the LLOQ concept where appropriate.

The Agency refined the procedure for establishing the LLOQ. This refinement considers sample matrix effects; includes a provision to verify the reasonableness of the reported quantitation limit (QL); and recommends a frequency of LLOQ verification (found in Chapter One and each method) to be balanced between rigor and practicality.

The Agency understands that previous versions of methods published in SW-846 may contain the MDL reference and as methods are updated, the Agency will remove references to the MDLs. The Agency will also remove MDL references in older methods that have not yet been

updated, as time and resources allow. References in MDPs will be revised in a future effort since they can only be revised through a notice and comment rulemaking effort. The Agency recommends the use of LLOQ, as appropriate, for the non-MDP methods that have not yet been updated. See Section 9.8 in Method 6020B for Inorganic analytes and Section 9.7 in Method 8000 for Organic analytes on LLOQ for further information on implementation. Also, if method users choose to run the LLOQ sample, it must be run with each batch to see if it meets the established acceptance criteria. Lastly, results above the LLOQ are quantifiable within an acceptable precision and bias. Thus, the LLOQ approach better suits the needs of the RCRA program, because it provides reliable and defensible results, especially at the lower level of quantitation, and can be reported with a known level of confidence for the complex matrices being evaluated. Various programs use SW-846 methods in implementing different statutes, including RCRA, the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA), the Toxic Substances Control Act (TSCA), the Oil Pollution Act, Homeland Security Presidential Directives and Presidential Policy Directives, for waste and materials characterization, compliance testing, site/incident characterization and extent of contamination, risk assessment, and remediation for protection of human health and the environment, and better management and use of wastes and materials, for a wide range of difficult matrices. The Agency believes that the LLOQ approach is an important improvement and supports the essential need to provide data that are verified to meet the precision and accuracy requirements of the RCRA program.

*Establishing the LLOQ for Inorganic Analytes* - When performing methods for inorganic analyses, the LLOQ should be verified by the analysis of at least seven replicate samples (prepared in a clean matrix or control material) and spiked at the LLOQ and processed through all preparation and analysis steps of the method. The mean recovery and relative standard deviation (RSD) of these samples provide an initial statement of precision and bias at the LLOQ. In most cases, the mean recovery should be no more than  $\pm 35\%$  of the true value and the RSD should be  $\leq 20\%$ . Ongoing LLOQ verification, at a minimum, is on a quarterly basis to validate quantitation capability at low analyte concentration levels. This verification may be accomplished either with clean control material (e.g., reagent water, method blanks, Ottawa sand, diatomaceous earth, etc.) or a representative sample matrix free of target compounds. Optimally, the LLOQ should be less than the desired regulatory action levels based on the stated project-specific requirements. For more information, please see the individual methods (e.g., Methods 6010 and 6020) and Chapter One of SW-846.

*Establishing LLOQ for Organic Analytes* - When performing methods for organic analyses, the LLOQ should be verified using either a clean control material (e.g., reagent water, method blanks, Ottawa sand, diatomaceous earth, etc.) or a representative sample matrix free of target compounds. Optimally, the LLOQ should be less than the desired regulatory action levels based on the stated project-specific requirements.

For organic analyses, the acceptable recovery ranges of target analytes will vary more than for other types of analyses, such as inorganics. The recovery of target analytes in the LLOQ check

sample should be within established limits, or other such project-required acceptance limits, for precision and bias to verify the data reporting limits. Until the laboratory has sufficient data to determine acceptance limits statistically, the laboratory control sample (LCS) criterion, +20% (i.e., lower limit minus 20% and upper limit plus 20%) may be used for an acceptable range for the LLOQ. This approach acknowledges the poorer overall response at the low end of the calibration curve. Historically based LLOQ acceptance criteria should be determined as soon as practical once sufficient data points have been acquired.

In-house limits (which a laboratory establishes) for bias (e.g., % Recovery) and precision (e.g., Relative Percent Difference (%RPD)) of the LLOQ for a particular sample matrix may be calculated when sufficient data points exist. The laboratory should have a documented procedure for establishing its in-house acceptance ranges. Sometimes the laboratory instrument and/or analyst performance vary or test samples cause problems with the detector (e.g., samples may have interferences; may clog the instruments cells, wall or tube; may cause contamination; etc.). Therefore, a laboratory establishes the limits of acceptance (for precision and bias) with sufficient data to demonstrate that they can report down to the LLOQ with a certain level of confidence. As an alternative, a QAPP may include the acceptance limits (for precision and bias) for LLOQ at the project level through the DQOs it includes. The frequency of the LLOQ check is not specified for organic analytes.

Note: The LLOQ check sample should be spiked with the analytes of interest at the predicted LLOQ concentration levels and carried through the same preparation and analysis procedures as environmental samples and other QC samples. For more information, please see individual

methods (e.g., Method 8000) and Chapter One of SW-846.

*Use of the LLOQ* - The RCRA program deals with complex wastes and materials that are managed or used in many different ways (e.g., landfilling, land application, incineration, recycling). The thresholds (e.g., action or clean up levels) for data users (e.g., engineers or risk assessors) to make their decisions, therefore, vary. Method users will need to properly plan their analytical strategy to ensure the LLOQs for targeted analytes are lower than the thresholds needed to generate data used to determine how waste or materials can be properly managed or used.

*Initial Demonstration of Performance (IDP)* - The IDP serves as a procedure that the laboratory conducts to demonstrate the ability to generate results with acceptable accuracy and precision for each preparation and determinative method they perform. Detailed discussion can be found in the October 23, 2013 Federal Register notice.

The Agency did not receive any comments on the IDP, and has finalized the language as presented in the original notice. Language regarding the IDP has been specified in the individual Update V methods where appropriate (e.g. Methods 6010D, 6020B, 8000D and many others).

The IDP changes allow laboratories to use their time and resources effectively, especially for the organic analyses. The IDP section for the Determination of Organic Analytes was expanded to describe two situations: when a significant change to instrumentation or procedure occurs: Reliable performance of the methods depends on careful adherence to the instructions in the written method because many aspects of the method are mandatory to ensure the method performs as intended.



Therefore, if a major change to the sample preparation procedure is made (e.g., a change of solvent), the IDP must be repeated for that preparation procedure to demonstrate the laboratory technician's continued ability to reliably perform the method. The Agency considers conducting IDPs as part of good laboratory practice procedures and has already included these procedures in the Agency's laboratories' practices. Alterations in instrumental procedures only (e.g., changing Gas Chromatograph (GC) temperature programs or High Performance Liquid Chromatography (HPLC) mobile phases or the detector interface), require a new calibration, but not a new IDP because the preparation procedure is unchanged.

When new staff members are trained: A new analyst needs to be capable of performing the method, or portion of the method, for which he/she is responsible. For example, when analysts are trained for a subset of analytes for an 8000 series method, the new sample preparation analyst should prepare reference samples for a representative set of analytes (e.g., the primary analyte mix for Method 8270, or a mixture of Aroclor 1016 and 1260 for Method 8082) for each preparation method the analyst will perform. The instrument analyst being trained will need to analyze the prepared samples (e.g., semi-volatile extracts). After several training opportunities, the analyst will be expected to perform the preparation and determinative step on his/her own and meet the acceptable QA/QC criteria.

*Blank Contamination* - Another area that affects sample results and is expanded upon in this notice and addressed in Chapter One and the individual methods is blank contamination. The results from analyzing blanks are generally considered to be acceptable if target analyte

concentrations are less than  $\frac{1}{2}$  the LLOQ or are less than project-specific requirements. Blanks may contain analyte concentrations greater than acceptance limits if the associated samples in the batch are unaffected (i.e., targets are not present in samples or sample concentrations are  $\geq 10\times$  the blank). Other criteria may be used depending on the needs of the project. For method specific details see Methods 6010 and 6020 for inorganics and Method 8000 for organics.

*Relative Standard Error (RSE)* - The Agency included RSE as an option (in addition to calculation of the % error) in Update V of SW-846 for the determination of the acceptability for a linear or non-linear calibration curve. The Agency received several comments from two commenters on RSE. The Agency agrees that Method 8000D, Section 11.5.6.1 on RSE should not be grouped with RSD and  $r^2$  (Regression Coefficient) but with % Error. Standard deviation (SD) and  $r^2$  are indicators for checking the validity of different calibration methods of response factor and least square linear regression techniques, respectively. RSE is not equivalent or similar to RSD or  $r^2$ , but similar to % Error and may be used to evaluate the “goodness of fit” of a calibration curve.

To avoid confusion with RSD, RSE has been moved to Section 11.5.4.2 of Method 8000D. In addition, the first sentence in Section 11.5.6.1 of Method 8000D has been changed to read as follows: "Corrective action may be needed if the calibration criteria (RSD/ $r^2$  and % Error/RSE) are not met." Some corrective actions may include running a new calibration, preparing fresh standards or performing instrument maintenance. The laboratory's SOPs should address how to handle and document these types of problems when encountered.

RSE refits the calibration data back to the calibration model and evaluates the difference between

the measured and the true amounts or concentrations used to create the model.

$$RSE = 100 \times \sqrt{\sum_{i=1}^n \left[ \frac{x'_i - x_i}{x_i} \right]^2 / (n - p)}$$

Where:

$x_i$  = True amount of analyte in calibration level  $i$ , in mass or concentration units.

$x'_i$  = Measured amount of analyte in calibration level  $i$ , in mass or concentration units.

$p$  = Number of terms in the fitting equation (average = 1, linear = 2, quadratic = 3, cubic = 4)

$n$  = Number of calibration points.

The RSE acceptance limit criterion for the calibration model is the same as the RSD limit in the determinative method.

If the RSD limit is not defined in the determinative method, the RSE limit should be set at  $\leq 20\%$  for good performing compounds and  $\leq 30\%$  for poor performing compounds.

### **Chapter Two (Choosing the Correct Procedure)**

The Agency received 12 comments on Chapter Two. Most comments were favorable, and others were editorial in nature. Therefore, the Agency has revised and finalized the Table of Contents to add the new and revised methods from Update V to the SW-846 compendium. Method titles from the 8000 series were added to Section 2.2.3 for completeness. Other tables were revised to include additional analytes as appropriate. In addition, a typographical error for bis(2-chloroisopropyl) ether was corrected to bis(2-chloro-1-methylethyl) ether in Tables 2-1, 2-4, 2-

15, 2-22, and 2-34. This correction is consistent with the most common way to identify this compound. New compounds were also added to Tables 2-1, 2-6, 2-20, 2-23A, 2-29A, 2-30, 2-31, 2-35A, 2-36A, 2-41, 2-45 and 2-46. Furthermore, Table 2–40(A) includes the current sample preservation guidance for styrene and vinyl chloride in aqueous samples (i.e., deletion of previously recommended practice of collecting a second set of samples without acid preservatives and analyzing immediately, if styrene and vinyl chloride are analytes of interest), and Table 2–40(B) includes Mercury Speciation hold times in addition to totals. Figure 2-2 was updated to include the most up-to-date guidance and to streamline the flowchart.

### **Chapter Three (Inorganic Analytes)**

The Agency received six comments on Chapter Three. Most comments were favorable, and the Agency made the appropriate editorial and clarification changes (e.g., removed reference to trip blank in Section 3.3.2, title change to Table 3-2 Digestion Volume/Mass, etc.). The change included finalizing the revised definition for Instrument Detection Limit (IDL) to be consistent with the revised Methods 6010D and 6020B. In addition, the term “bias” has replaced “accuracy” where appropriate; the definition for linear range is now consistent with Methods 6010D and 6020B. The definition for the spectral interference check (SIC) solution has replaced the definition for the interference check sample (ICS) and is consistent with Methods 6010D and 6020B. The definition of LCS (laboratory control sample) recommends the use of a spiking solution from the same source as the calibration standards. Sections 3.6 and 3.7 were finalized to include the collision/reaction cell technology as an effective method for removing isobaric interferences when analyzing by ICP–MS. Table 3–2 now includes a minimum mass of 100 g for

solid samples collected for sulfide analysis.

#### **Chapter Four (Organic Analytes)**

The Agency received nine comments on Chapter Four. Most comments focused on Table 4–1, which has now been finalized to exclude the recommendation to collect a second set of samples without adding an acid preservative and analyze in a shorter time frame if vinyl chloride and styrene are analytes of concern for aqueous samples. A study showed that there were no significant differences in sample recovery of those samples preserved with acid versus those not preserved. Other comments were minor, and appropriate revisions have been made adding additional methods to section 4.3.3.

#### **Chapter Five (Miscellaneous Test Methods)**

The Agency did not receive any comments on Chapter Five. Chapter 5's changes were general (i.e., updated format changes and method reference to chapters), and it was finalized as appropriate.

#### **Chapter Nine (Sampling Plan)**

The Agency also received comments on Chapter Nine, which was not open for comment. However, the Agency will consider those comments in a future update.

### ***B. Methods Revisions***

Significant revisions were finalized regarding Methods 6010D, 6020B, and 8000D, and are discussed in this notice. Many methods were revised based on technical and editorial comments

received during the comment period. More detailed discussions and responses to all comments received on Update V can be found in the Response to Comments Background Document in the RCRA Docket at: (EPA-HQ-RCRA-2012-0072). A summary of significant comments has been provided.

*Method 6010D (Inductively Coupled Plasma – Atomic Emission Spectrometry)* - The Agency received 12 comments on Method 6010D. Most comments were favorable and applauded consistency revisions between methods and chapters. Several commenters requested that the guidance should clarify how to establish the LLOQ for inorganic methods in instances when regulatory limits are much lower than the lowest calibration standard. In response, the Agency added language to address the reporting of flagged data and other options in interpreting data when the desired LLOQ has not been met. In addition, revisions were made where technical and editorial comments were appropriate (e.g., title changes and relevant information specific to inorganics or organics). See section 9.8 of the method for more information on interpreting the LLOQ.

In addition, the Agency received other comments regarding clarification of the method blank acceptance criteria and definitions (such as Instrument Detection Limit procedure (IDL)) which can be found in detail in Method 6010D.

*Method 6020B (Inductively Coupled Plasma-Mass Spectrometry)* – The Agency received nine comments on Method 6020B. Many comments pertained to the Initial Calibration Blank (ICB), when multi-calibration standards are used, and the LLOQ. The Agency agreed with the commenter and revised the appropriate section in Method 6020 to read as follows: “If the ICB

consistently has target analyte concentrations greater than half the LLOQ, the LLOQ should be re-evaluated.” In addition, the Agency has clarified the statement that if there is no regulatory limit and the method blank is >10% of the lowest sample concentration, then the method blank may be considered to be acceptable if <LLOQ. In addition, typographical errors were corrected.

*Method 8000D (Determinative Chromatographic Separations)* – The Agency received comments on Method 8000 during the public comment period and an additional four afterward. The comments received are summarized below in several categories.

Eight comments were related to the use and implementation of the LLOQ and its application to method blanks. Several additions and changes were made in the method as a result of these comments. The method blank language in Sections 9.2.6.9 through 9.2.6.11 was updated to reflect that blanks should be considered acceptable if the concentrations found were below one half of the LLOQ (or project DQOs). Blanks may contain hits for reported compounds if the results in the associated samples are >10X the concentration in the blank. The data may also be reported with flags, which is a new option in this version of Method 8000.

Seven comments were related to QC sample frequency and control limits. One commenter requested that a numerical limit for LLOQ standard recovery be used. The users are encouraged to develop statistical acceptance limits rather than to default to a set of numerical limits in the method. The suggested criteria remain  $\pm 20\%$  of the laboratory's control sample (LCS) limits. Another commenter objected to removal of the word “must” from some calibration criteria (such as calibration coefficients). The Agency confirmed the intention to allow the project requirements to be flexible. The laboratories are also instructed to perform corrective actions

whenever calibration criteria for their project requirements are not met. Some other suggestions were not adopted (such as a requirement to run an end continuing calibration verification (CCV) for every 8000 series method or to require all extraction QC from a batch to be run on the same instrument as every sample and/or dilutions thereof). The Agency's view is that the methods should remain flexible and more restrictive QC requirements (where needed) should be listed in the determinative methods.

One commenter requested the inclusion of an additional reference (the Department of Defense Quality Systems Manual, Version 5.0 (DOD QSM 5.0)) as The Agency used it in developing Update V. The Agency agrees, and added the reference.

*Methods 8021B (Aromatic and Halogenated Volatiles by Gas Chromatography Using Photoionization and/or Electrolytic Conductivity Detectors), 8111 (Haloethers by Gas Chromatography), and 8430 (Analysis of Bis(2-chloroethyl) Ether and Hydrolysis Products by Direct Aqueous Injection GC/FT-IR)* – The Agency received the same two comments for these three methods. Both comments concurred with the nomenclature change for bis(2-chloro-1-methylethyl)ether, which alleviated confusion.

*Method 8270D (Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS))* – The Agency received two comments which concurred with the nomenclature change for bis(2-chloro-1-methylethyl)ether. Method 8270D also received one comment asking about the possibility of reporting flagged data from calibrations where some compounds were outside the specified criteria. The Agency's RCRA Organic Workgroup is discussing this issue and intends to address it in Update VI.



*Method 8410D Gas Chromatography/Fourier Transform Infrared (GC/FT-IR) Spectrometry for Semivolatile Organics: Capillary Column* – The Agency received two comments on Method 8410D which concurred with the nomenclature change for bis(2-chloro-1-methylethyl)ether. Method 8410D also received one comment discussing the acceptable temperature range of samples for preservation. The Agency accepted the updated change.

*Method 9014 (Titrimetric and Manual Spectrophotometric Determinative Methods for Cyanide)* – Detailed information on calibration models and their acceptance criteria are not included in each SW-846 method. This is because these methods are intended as general guidance, as are all of the methods discussed in this notice. For any test method which is not a method-defined parameter (MDP), the intention is to allow the laboratory flexibility under the Methods Innovation Rule (MIR).<sup>3</sup> The details of how a laboratory will conduct and approve calibrations should be included in the individual laboratory's Quality Management Plan (QMP) or in its Standard Operating Procedure (SOP) for each method.

*Method 9040 (pH Electrometric Measurement)* – This method is a Method Defined Parameter (MDP) and the Agency cannot revise an MDP through a Notice of Availability, but instead must use notice-and-comment rulemaking procedures. During a future rulemaking effort, the Agency will consider those comments on MDPs that may require rulemaking.

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<sup>3</sup> See 70 FR 34537, June 14, 2005 Federal Register.

## **V. Summary**

These changes in Update V will assist method users in demonstrating method competency and in generating better quality data. For the convenience of the analytical community, the Agency will revise the OSWER Methods' Team homepage on The Agency's web site to include the final Update V. Also, please see the web site:

<http://www.epa.gov/epawaste/hazard/testmethods/index.htm> for more information. Table 1 provides a listing of the five chapters and 23 methods (8 new methods and 15 revised methods) in Update V.

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Office of Resource Conservation and Recovery.

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